FIBER DAMAGE DURING THE CONSOLIDATION OF PVD Ti-6Al-4V COATED NEXTEL 610™ ALUMINA FIBERS

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(Received 22 December 1994; in revised form 16 February 1995)

Abstract—Titanium matrix composites reinforced with sol–gel synthesized α-alumina fiber tows have attracted interest as a potentially low cost continuous fiber reinforced metal matrix composite system. We have conducted a detailed investigation of fiber damage during high temperature consolidation of PVD Ti-6Al-4V metallized sol–gel alumina fiber tows. Using both hot isostatic pressing and interrupted vacuum hot press consolidation cycles, the two principal mechanisms of fiber damage have been experimentally identified to be microbending/failure and fiber matrix reaction. A time dependent micromechanics model incorporating the evolving geometry and mechanical properties of both the fibers and matrix has been formulated to simulate the fiber bending/failure mechanism in a representative unit cell and explore the effect of fiber strength loss due to reaction with the matrix. This model has been used to design a process cycle that minimizes damage by exploiting the enhanced superplastic deformation of the initially nanocrystalline PVD Ti-6Al-4V matrix.

1. INTRODUCTION

Intensive efforts are underway to develop low cost, continuous fiber reinforced titanium matrix composite (TMC) systems. One approach seeks to utilize inexpensive sol–gel synthesized NEXTEL 610™ α-alumina fiber tow consisting of 112, 12 µm diameter fiber filaments [1]. A metal matrix composite may be manufactured from this fiber system by thinly coating all the fibers in a tow with an interphase coating which protects the fiber from chemical reaction with matrix alloys during both high temperature processing and use. A thicker layer of physical vapor deposited (PVD) matrix material (for example Ti-6Al-4V) is then deposited using a PVD process such as sputtering or electron beam evaporation. These “metallized” fiber tows are then packed in either hot isostatic press (HIP) or vacuum hot press (VHP) tooling and subjected to a high temperature consolidation cycle resulting in a near net shape, fully dense TMC component. We report here on the potential fiber damage mechanisms accompanying this manufacturing approach.

The usual starting point for designing a composite consolidation cycle is that used for monolithic matrix alloy powders. These usually consist of a rapid application of stress (or pressure) with simultaneous heating so as to maximize the pressurization rate and reach a preselected consolidation stress and temperature in as short a time as possible [2, 3, 4]. This is then followed by an “isothermal soak” to promote time dependent (creep) deformation until the desired density is reached. When processing metal matrix composite parts by the deformation processing methods outlined above, consideration must also be given to a number of factors, including attaining full matrix density, avoiding fiber fracture, and maintaining the strength of the fibers (i.e. avoiding degradation by chemical reactions with the matrix).

Under ideal conditions straight circular cross-section fibers uniformly metallized over their circumference can be packed in a hexagonal close packed (h.c.p.) structure with an initial relative density of 0.906 (volume fraction). In this case, provided the matrix–fiber thermal expansion mismatch does not cause large tensile fiber stress on heating, and precautions are taken to eliminate the possibility of axial matrix flow near the ends of fibers (and thus induce tensile fiber loading), the close-packed fiber arrays are relatively simple to consolidate to full density with minimal risk of fiber bending or fracture. However in metal coated fiber tows, deviations from the close packed structure can occur due to (a) fiber misalignment (e.g. crossing fibers), (b) nonuniform thickness of the fiber coating, (c) variations in metallized fiber diameter or (d) clustering of fibers during their fabrication (see Fig. 1). Such “defects” are responsible for aberrations in the packing and the existence of large voids, which lower the specimen’s initial packing density.

Processing to full density (without fiber fracture) might still be achievable if both sufficient matrix material is present near the voids and consolidation conditions are chosen such that matrix material can infiltrate the voids. The alternate, highly undesirable, densification mechanism is fiber bending into the void
which, when excessive, leads to fiber fracture. Even fiber microbending without fracture can be deleterious if cracking or spalling of the protective interphase fiber coating occurs, thus locally exposing the fiber to the titanium matrix. When this occurs at the high processing temperatures typically used, the high chemical reactivity of titanium alloy matrices is likely to result in serious degradation of the fiber's strength [5]. Even when fiber fracture or strength loss do not occur, the locked-in residual stresses of bent fibers may combine with stresses applied during service, resulting in a potentially significant loss of composite strength [6].

Determining, or even ascertaining the existence of, the optimum process cycle for a system of this complexity typically requires a multitude of consolidation cycles and time consuming fiber damage characterization studies [7, 8, 9]. Rather than resorting to this trial and error approach, here we explore an alternative strategy in part motivated by the limited supply of these materials for testing. A few consolidation experiments are conducted and analyzed to determine the mechanisms responsible for fiber damage. The experiments will show that conventional hot consolidation processing cycles, developed for powder densification, are unsuitable for densifying metallized fiber tows. A micromechanical model (corresponding to the mechanisms of damage) is then developed which predicts the dependence of fiber bending and fracture on lay-up geometry, material (fiber-matrix) properties and process conditions. The model is then used to design a high temperature process cycle that minimizes fiber damage and further experiments conducted to verify its predictions.

2. THE MATERIAL SYSTEM

The composite material system investigated here was synthesized at the 3M Metal Matrix Composite Center (Mendota Heights, Minn.). It consisted of 10 m of metallized NEXTEL 610™ alumina fiber tow. Each fiber in the tow was ~12 µm in diameter and had been coated first with a protective interphase layer and then a thicker layer of Ti-6Al-4V (see Fig. 1). The initial relative density of the tows when packed into the dies used for consolidation was between 0.45 and 0.55. When fully consolidated, the resulting TMC system contained approx. 0.35 volume fraction of fiber.

2.1. NEXTEL 610™ α-alumina fiber

The continuous NEXTEL 610™ α-alumina (>99.3% α) fibers were synthesized by a sol-gel technique from a mixture of aluminum carboxylate sol precursors [1]. A tow consisting of 112 precursor fibers was formed in a single spinning operation. This spinning process resulted in a small fraction of non-aligned fibers. These “green” filament tows were then annealed (at low temperature) to remove volatile components (via a pyrolysis process) producing weak amorphous aluminum oxide fiber tow. A subsequent high temperature heat treatment was then used to crystallize the amorphous oxide, and produce a nanocrystalline (<0.1 µm grain size) α-alumina microstructure fiber of high ambient temperature strength [10] (Table 1).

2.2. Interphase coating

A three-layer protective interphase coating was deposited on the bare fiber to inhibit fiber-matrix reactions during the high temperature consolidation step [11]. First, a 50–100 nm thick boron nitride (BN) layer was deposited directly onto the bare fiber surface. This was followed by a 100 nm thick carbon coating and finally a 600–800 nm thick layer of titanium diboride (TiB2). Each layer was deposited by chemical vapor deposition (CVD). The BN layer protected the fiber from the volatile precursors evolved during the subsequent deposition of the TiB2 coating. The carbon coating served to both enhance TiB2 surface nucleation and to act as a low sliding stress layer between the fiber and the matrix [12].

2.3. Matrix coating

The Ti-6Al-4V matrix was directly deposited onto the interphase coated fibers using an Ar plasma sputtering PVD method (background pressure ~2 × 10^-4 torr) with a deposition rate of approx. 0.8 µm/s. The surface of the fiber was estimated to be between 250 and 300°C during deposition. In the
as-deposited condition the matrix consisted of metastable (h.c.p.) α- and martensitic (h.c.p.) α'-phases with an average grain size between 50 and 100 nm. It was similar to that analyzed in Ref. [13]. When heated to 900°C and held at this temperature for 1 h the matrix material transformed into a two phase, α + (b.c.c.)β, microstructure with an average grain size of ~2 μm. The volume fraction of each phase and its grain size were time and temperature dependent and hence changed during the consolidation processing. The microstructural evolution and the resulting high temperature, time dependent creep behavior of this matrix has been previously characterized [13]. The alloy was found to deform by a significantly enhanced superplastic mechanism with measured strain rates between 3 and 10 times those observed in conventionally processed Ti-6Al-4V at similar temperatures (760–900°C) and stresses (5–60 MPa). The enhanced superplastic behavior was shown to be consistent with the refined grain size of the PVD processed matrix [13].

The stress–strain rate response for this superplastic matrix is well approximated by a semi-empirical relationship [14, 15]

$$\dot{\varepsilon} = B_0\exp\left[\frac{-Q}{RT}\right] \frac{\sigma^n}{d(t)^p}$$

(1)

where \(\dot{\varepsilon}\) is the strain rate, \(\sigma\) is the applied stress, \(n\) is the creep stress exponent, \(B_0\) is a temperature independent creep constant, \(d(t)\) is a (consolidation) temperature and time dependent grain size, \(p\) is the grain size dependence coefficient (which can vary between 1 and 3 depending upon the strain rate and temperature), \(Q\) is an activation energy for superplastic flow, \(R\) is the universal gas constant and \(T\) the absolute temperature. The grain size evolution, \(d(t)\), determined experimentally for the consolidation temperatures used here, can be described by

$$d(t) = d_0 + k(t)^a$$

(2)

where \(d_0\) is the initial grain size, and \(k\) and \(a\) are temperature dependent experimentally determined constants. The material parameters in equations (1) and (2) for this matrix are summarized in Table 1.

### Table 1. Material properties for the NEXTEL 610M-PVD Ti-6Al-4V composite system

<table>
<thead>
<tr>
<th>Fiber properties</th>
<th>Symbol</th>
<th>Value</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fiber diameter (μm)</td>
<td>(d)</td>
<td>12 ± 1</td>
<td>[10]</td>
</tr>
<tr>
<td>Fiber Young's modulus (MPa)</td>
<td>(E_t)</td>
<td>390,000</td>
<td>[10]</td>
</tr>
<tr>
<td>Fiber reference strength (MPa)</td>
<td>(\sigma_f)</td>
<td>2380</td>
<td>[10]</td>
</tr>
<tr>
<td>Coefficient of thermal expansion (°C⁻¹)</td>
<td>(\alpha)</td>
<td>(9.5 \times 10^{-6})</td>
<td>[27]</td>
</tr>
<tr>
<td>Fiber Weibull modulus</td>
<td>(m)</td>
<td>9</td>
<td>[10]</td>
</tr>
<tr>
<td>Fiber volume fraction</td>
<td>(V_f)</td>
<td>0.35</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Matrix Properties</th>
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<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature independent creep parameter (m (MPa)⁻ⁿ s⁻⁻)</td>
<td>(B_0)</td>
<td>0.000004</td>
<td>[13]</td>
</tr>
<tr>
<td>Creep stress exponent</td>
<td>(n)</td>
<td>1.3</td>
<td>[13]</td>
</tr>
<tr>
<td>Activation energy for superplastic flow (kJ mol⁻¹)</td>
<td>(Q)</td>
<td>150</td>
<td>[13]</td>
</tr>
<tr>
<td>Grain size exponent</td>
<td>(p)</td>
<td>1</td>
<td>[13]</td>
</tr>
<tr>
<td>Grain growth exponent at 840°C</td>
<td>(a)</td>
<td>0.20</td>
<td>[13]</td>
</tr>
<tr>
<td>Grain growth constant at 840°C (μm s⁻⁻)</td>
<td>(k)</td>
<td>0.23</td>
<td>[13]</td>
</tr>
<tr>
<td>Initial grain size at 840°C (μm)</td>
<td>(d_0)</td>
<td>0.8</td>
<td>[13]</td>
</tr>
<tr>
<td>Coefficient of thermal expansion (°C⁻¹)</td>
<td>(\alpha_v)</td>
<td>(11.8 \times 10^{-6})</td>
<td>[28]</td>
</tr>
<tr>
<td>Matrix volume fraction</td>
<td>(V_m)</td>
<td>0.65</td>
<td></td>
</tr>
</tbody>
</table>

*Prior to consolidation processing.

Ambient temperature grain size is between 50 and 100 nm.

Value is for elemental titanium.

The stress–strain rate response for this superplastic matrix is well approximated by a semi-empirical relationship [14, 15]

$$\dot{\varepsilon} = B_0\exp\left[\frac{-Q}{RT}\right] \frac{\sigma^n}{d(t)^p}$$

where \(\dot{\varepsilon}\) is the strain rate, \(\sigma\) is the applied stress, \(n\) is the creep stress exponent, \(B_0\) is a temperature independent creep constant, \(d(t)\) is a (consolidation) temperature and time dependent grain size, \(p\) is the grain size dependence coefficient (which can vary between 1 and 3 depending upon the strain rate and temperature), \(Q\) is an activation energy for superplastic flow, \(R\) is the universal gas constant and \(T\) the absolute temperature. The grain size evolution, \(d(t)\), determined experimentally for the consolidation temperatures used here, can be described by

$$d(t) = d_0 + k(t)^a$$

where \(d_0\) is the initial grain size, and \(k\) and \(a\) are temperature dependent experimentally determined constants. The material parameters in equations (1) and (2) for this matrix are summarized in Table 1.

### 3. PACKING CHARACTERISTICS OF METALLIZED FIBER TOWS

The consolidation of metallized fiber tows is a sensitive function of the initial packing geometry (i.e. the initial density and the fiber alignment distribution). The initial packing density of three samples was measured by placing metallized fibers in channel dies, impregnating with epoxy and then lightly compacting. After allowing the epoxy in these “preconsolidated samples” to cure, they were cross-sectioned and polished for metallographic analysis to determine the density (taken to be the ratio of coated fiber to total compact cross-sectional area). The measurements indicated that the tows typically packed to an initial density of ~0.50 before the applied load becomes high enough to cause permanent densification. This is significantly lower than the ~0.91 relative density attained by the 2D hexagonal close-packing of parallel perfectly straight cylinders. The low initial packing density of the preconsolidated specimens here was principally caused by fiber crossover (i.e. fiber misalignment); small additional contributions may be attributed to nonuniformity of the metallized coating (i.e. clustering of fibers during matrix deposition), fiber diameter variations and fiber curvature due to differential cooling strains in nonuniformly coated samples.

Figure 2 schematically represents the fiber packing configuration showing fiber crossing (the crossover angle has been exaggerated to more clearly illustrate the bending geometry). The crossover contacts are assumed to be matrix–matrix contacts; the interweaving fiber spanning the contacts is seen to be unsupported and is therefore susceptible to bending.
during consolidation. The initial packing configuration can be characterized by the distribution of separation distances between adjacent crossover contacts. This distribution has been obtained from a quantitative metallographic analysis of preconsolidated specimens using a simple geometric relationship between the angle of fibers, their spacing and the intercontact length, Fig. 3(c). If the intercontact

![Diagram](image-url)

**Fig. 2.** A schematic representation of the type of packing associated with this composite material system.

**Fig. 3.** Simple geometric relationship used to determine the distribution of fiber span lengths in a metallized fiber compact prior to consolidation (see text).
length, in a plane normal to the consolidation load is \( s_n \), and the magnitude of the fiber span angle \( \varphi_p \), then the separation distance between contacts is

\[
I_{qr} = \frac{s_n}{\sin(\varphi_p)}.
\]

(3)

Both the nearest neighbor spacing, \( s_n \), the fiber span angle \( \varphi_p \), are statistically distributed with probability distributions \( P_1(s_n) \) and \( P_2(\varphi_p) \). If we assume \( P_1(s_n) \) and \( P_2(\varphi_p) \) are statistically independent, the probability distribution for the intercontact length, \( l_{qr} \), is

\[
P[l_{qr}] = P_1(s_n)P_2(\varphi_p).
\]

(4)

The nearest neighbor fiber spacing, \( s_n \), and its frequency of occurrence was determined from metallographic cross-sections. Four epoxy-impregnated, preconsolidated fiber compacts, packed to initial relative densities between 0.45 and 0.55 were examined in a scanning electron microscope (SEM). For each cross-section, micrographs were taken and assembled into a large portrait containing approx. 660 fiber ends. A series of equally spaced horizontal lines were then laid over each portrait, individual separation distances between nearest neighbor fibers along each line measured and a distribution of separation distances obtained, Fig. 4(a).

A measure of the angular distribution of metallized fibers in the preconsolidated state was obtained by packing the tows in a fixture which simulated a VHP compression die. The fixture allowed SEM observations to be made through a small window located in the plane of the packed tows. SEM micrographs were taken over a \( 4 \times 4 \) mm (4 mm \( \approx 254 \) d) area within the opening, and assembled into a single portrait. The angular distribution was obtained by direct measurement using a drafting instrument specifically designed to measure angles. Four separate samples were measured and the average is shown in Fig. 4(b). The resulting probability distribution of contact separation distances, \( P[l_{qr}] \) vs \( l_{qr} \), obtained using equations (3) and (4) is shown in Fig. 4(c). Figure 4(c) reveals that approx. 90% of the fibers crossover adjacent fibers. If fiber fracture occurs during consolidation, a distribution of fiber segments, comparable to Fig. 4(c), should result.

4. EXPERIMENTAL PROCEDURES

4.1. Consolidation method

Consolidation experiments were conducted in both a VHP and HIP. The VHP tests enabled densification data to be obtained with relatively simple instruments, avoiding the complexities of fabricating, welding and removing HIP consolidation canisters, and enabled process loads to be more precisely defined and controlled.

VHP consolidation studies were conducted using a specially designed, molybdenum alloy (TZM), compression fixture capable of applying controlled load increments to 45 kN at test temperatures up to 1000°C. For this study a rectangular consolidation die \( 75 \times 7.5 \) mm was located between the compression platens. The consolidated thickness of the specimen (which varied with density) ranged between 0.6 and
The VHP compression fixture and load train were contained within a hermetically sealed, water-cooled, alumina retort attached to a vacuum pumping station (the vacuum environment, maintained at \(\sim 1 \times 10^{-4}\) torr during consolidation, was necessary since both the Ti-6Al-4V matrix alloy and TZM molybdenum hot pressing apparatus will oxidize rapidly in air at temperatures above 700°C). A programmable "clam-shell" type furnace surrounded the retort. Platen displacement, and hence instantaneous specimen height, was monitored using a linearly variable capacitive transducer (LVCT) system located in a "cold zone" in the retort assembly. Since the average specimen density is linearly proportional to its height recording the instantaneous height of the specimen during the consolidation cycle (minus the compression fixtures' elastic strain), allowed the instantaneous density to be determined provided the specimen height and density at the end of the cycle was also measured. Specimen temperature was measured using a K-type thermocouple mechanically attached to the upper compression platen. An in-line load cell was used to measure process loads and a strip chart recorder simultaneously collected data from the load cell and the LVCT unit. The specimen was loaded into the compression fixture, heated to the preselected consolidation temperature at a rate of 4°C/min and then compressed.

### 4.2. Fiber fracture characterization

The extent of fiber damage caused by the process cycle was determined by chemically removing the matrix material. Two sections, each 2.1 mm long (i.e. \(-180\) fiber diameters) were cut transversely to the fiber axis from each consolidated sample using a slow speed diamond wafering saw. These were then immersed in a dilute hydrofluoric (HF) acid solution (15 vol. % HF and a balance of distilled water) at ambient temperature. Care was taken to ensure the etching procedure did not introduce fiber damage. Similar high purity \(\alpha\)-alumina specimens exposed to room temperature solutions of 15% HF and 48% HF for 24 h periods have been reported to exhibit no detectable weight loss [16] (the resistance to aqueous HF corrosion for a wide range of glass and refractory ceramics is raised substantially by increasing the volume fraction of \(\alpha\)-alumina in the microstructure).

After a 1 h immersion, the matrix was completely dissolved, leaving fiber segments lying on a molybdenum plate placed in the bottom of the container prior to specimen immersion. After rinsing, the molybdenum plate and the fiber segments resting on it, were sputter-coated with Au prior to performing an SEM analysis to obtain the distribution of fiber segment lengths. Approximately 20 photomicrographs were taken (at a magnification of \(30 \times\) ) of a randomly selected area and assembled into a single large portrait. For each consolidation test, between 500 and 600 fiber segments were measured directly from each corresponding portrait.

The results of the fiber segment measurements were also expressed in terms of the number of fiber fractures per meter of fiber contained in the specimen by noting that the total length of fiber, \(L\), contained in the section removed from the consolidated specimen can be expressed as

\[
L = n_a + \sum_i n_i b_i
\]

where \(n_a\) and \(a\) are the number and length respectively, of unbroken fiber segments taken from the sample, and \(n_i\) and \(b_i\) are the number and length respectively of broken segments (broken segments have lengths less than \(a\)). The fiber segment lengths are discretized such that the subscript, \(i\), refers to the range in which a particular segment's length falls (fiber segments within a given range of length, for example 15–16 fiber diameters, would be considered the same length, \(b_i\)). For the sections removed here the unbroken length, \(a\), was equal to 2.1 mm.

To determine the number of breaks per meter of fiber note that when a single fiber of length \(a\) breaks into \(q\) segments it contains \((q - 1)\) fractures. If two fibers, each of length \(a\), break into \(q\) and \(r\) segments respectively the total number of fractures then becomes \([(q + r) - 2]\). The total number of fractures, \(n_f\), contained within the entire sample section is therefore

\[
n_f = \sum_i n_i - \left(\frac{\sum_i n_i b_i}{a}\right)
\]

where

\[
\sum_i n_i
\]

is the total number of broken segments counted and

\[
\left(\frac{\sum_i n_i b_i}{a}\right)
\]

represents the total number of fibers from which the segments were derived prior to becoming fractured. The number of fiber fractures per meter, \(\bar{n}_f\), obtained from equations (5) and (6) is then

\[
\bar{n}_f = \frac{1}{L} \left[\sum_i n_i - \left(\frac{\sum_i n_i b_i}{a}\right)\right]
\]

The mechanisms of fiber damage were investigated by partially macroetching the matrix of the consolidated specimen to expose the fiber reinforcement without removing the fiber from the specimen. The etched specimen was then placed in the SEM for analysis. These observations also revealed qualitative information about fiber orientation, nearest neighbor spacing and the condition of the fiber.
5. EXPERIMENTAL RESULTS

5.1. Baseline schedule

Figure 5(a) shows a HIP stress-temperature cycle which was used initially to fully consolidate the metallized fiber tows. This HIP cycle is similar to those used to consolidate Ti-based alloy powders [17] although in this case the pressure was applied after the sample had reached ~750°C. Figure 5(b) shows the resultant fiber segment distribution associated with this baseline process cycle which resulted in approx. 560 fractures/m. An unbroken fiber would have an \( l_f/d_f \) ratio of ~180, so it is evident that a large fraction of the fibers have been damaged. Metallography revealed that the majority of fiber fractures were due to bending (Fig. 6).

5.2. VHP process variations

Prior to conducting VHP experiments, tests were conducted to determine if fibers were damaged by the packing procedures or by the temperature cycle alone (without applied pressure) used for consolidation. A bundle of metallized fiber tow (consisting of 7840 metallized fibers each cut to a length of ~70 mm) was packed into a VHP die, inserted into the compression fixture and exposed to 1 h vacuum heat treatment at 840°C without a load applied. No broken fiber segments were found after subsequent matrix dissolution and it was concluded that fiber breaks were not caused by either packing or thermal processing.

To investigate the effect of the loading rate on fiber damage during the early stages of specimen consolidation, two different process cycles, VHP-1 and 2, were conducted at 840°C [see Figs 7(a) and 8(a)]. The cycle VHP-1 was designed using a model (described later) to have a loading rate that would not significantly fracture fibers. The VHP-2 cycle used a more rapid loading rate, typical of current practice.

The relative density as a function of time for the VHP-1 and 2 cycles is shown in Figs 7(b) and 8(b), respectively. Each cycle achieved a maximum density of 0.85. Fiber segment distributions are shown in Figs 7(c) and 8(c). When the process stress was applied gradually (VHP-1), the degree of process-induced fiber damage (120 fractures/m) was significantly lessened (VHP-2 had 600 fractures/m). We will show that the strong effect of loading rate on the fiber damage accumulated during consolidation can be rationalized by examining the force balance occurring between elastically bending fibers and creeping matrix contacts in a representative unit cell model (Section 6.2).

The influence of consolidation temperature on fiber damage was investigated by applying the process pressure to a new sample in a manner similar to that
of VHP-1, but at a higher temperature of 900°C for 1 h (designated as cycle VHP-3), Fig. 9(a). The sample had an initial density of about 0.45 [Fig. 9(b)] and, after consolidation, had reached a density of 0.83 which was somewhat lower than that measured for VHP-1 (0.85). The resultant fiber damage measured in VHP-3, Fig. 9(c), was however significantly lower (only 88 fractures/m) than the damage measured in VHP-1 (120 fractures/m) suggesting a beneficial effect of increasing the consolidation temperature. If the dominant fiber fracture mechanism at low relative densities is fiber bending, it would be reasonable to expect that the rate of fiber damage would approach zero as the density approached unity.

Fig. 7. (a) Consolidation cycle VHP-1, (b) measured relative density and (c) resultant fiber segment distribution.

Fig. 8. (a) Consolidation cycle VHP-2, (b) measured relative density and (c) resultant fiber segment distribution.
Thus at higher densities, $D > 0.85$, fiber breakage would be expected to be less sensitive to the rate of the applied processing pressure. To test this experimentally, a two-step process cycle [designated VHP-4, Fig. 10(a)] was conducted. It consisted of first an initial consolidation step similar to VHP-3 followed by a constant pressurization rate of $0.6 \, \text{MPa/min}$ (30 MPa/h) for a total cycle time of 2 h at 900°C. The cycle was terminated at a final pressure of 30 MPa and resulted in the specimen reaching a final relative density near 0.95.

The evolution of specimen density is shown in Fig. 10(b) and the fiber segment distribution is shown in Fig. 10(c). Approximately 176 fractures/m were...
measured for the VHP-4 cycle (twice the value measured for VHP-3) and suggests that fiber damage is less sensitive to the applied pressurization rate as the density increases. The reason for this may be that matrix creep fills void spaces in the compact limiting further fiber bending deflection and hence the fiber’s bending stress.

Figure 11(a) shows a cross-section of the VHP-4 specimen in which a fiber span, surrounded by a dense matrix region, has deflected into a void. A cross-section (normal to the fiber axis) is shown in Fig. 11(b). For this case, insufficient matrix material exists near the void and further densification can presumably occur only by fiber fracture.

5.3. Fiber chemical attack

SEM analysis of fiber segments removed from each specimen occasionally showed signs of fiber–matrix interactions (localized pitting), Fig. 12, and suggested a possible weakening of the fibers. The effect of the consolidation cycle on fiber strength has been determined by electrolytically extracting fibers from HIP-consolidated (at 900°C, 100 MPa) PVD coated fiber samples and testing them in tension [18]. The extracted fibers long enough to be tested had an average fracture strength of 1306 MPa compared to 2503 MPa for unprocessed fibers. Local defects in the fiber surface, such as those seen in Fig. 12, presumably reduce the fiber’s reference strength [19] and could contribute to increased fiber fracture during prolonged high temperature consolidation processing.

6. DISCUSSION

6.1. Damage mechanisms

Consolidation cycles subject fibers to both mechanical and chemical processes which can result in fiber damage. The extent of this damage may be severe, depending on the fiber–matrix-interphase materials and the process conditions used. One potential fracture mechanism could be due to the difference in coefficient of thermal expansion between the α-alumina fiber (9.5 × 10^-6 °C^-1 [20]) and the Ti–6Al–4V matrix coating (11.8 × 10^-6 °C^-1 [21]) which puts the fiber in axial tension during the heat-up stage of the consolidation cycle. The fiber’s axial stress, σ, due to this can be estimated from an elastic thermal stress model [22]:

$$\sigma = \frac{E_f \Delta \alpha \Delta T}{1 + V_f E_f (1 + V_m E_m)}$$

(8)

where $E_f$ and $E_m$ are the fiber and matrix Young’s moduli respectively, $V_f$ and $V_m$ are the fiber and matrix volume fractions respectively, $\Delta \alpha$ is the difference between the matrix and fiber thermal expansion coefficients, and $\Delta T$ is the difference between the deposition and the maximum processing temperature. Substituting the material property values from Table 1 into equation (8) the tensile axial fiber stress developed when heating to 900°C, ($\Delta T = 900°C$), is 262 MPa which is substantially lower than the average fracture strength of unprocessed fibers (2503 MPa). Since this elastic calculation overestimates the stress developed, exposure of metallized fiber tows to thermal cycles alone is thus not expected to cause significant fractures (see Section 4.2) and can be discounted.

At the onset of the consolidation cycle the initial packing density of the preconsolidated specimen was relatively low due to the inefficient packing of the metallized fiber tows. The structure of this low density
preform can be viewed as a three-dimensional, skeletal network of metallized fiber monofilaments, Fig. 2. When the low density fiber compact is compressed (at elevated temperature), the externally applied load is supported by the transmission of forces through the network of (matrix–matrix) crossover contacts. Fiber segments that are in bending configurations, Fig. 13, will therefore deflect under the influence of the applied load, leading to a break wherever the fibers strength limit is exceeded. This fiber bending appears to be the principal mechanical mechanism responsible for damage during consolidation.

Densification (or fracture) results from two competing mechanisms: (a) inelastic deformation of the matrix in the vicinity of contacts; and (b) elastic deflection of fibers into the void space between contacts. Minimizing the fiber bending stress and hence deflections requires that the loading and temperature be chosen to promote matrix flow at the contacts rather than bending of fibers. This postulate appears to agree with the experimental observations: (1) at low densities, fiber damage is strongly sensitive to the rate of applied loading; and (2) at higher densities damage is less dependent on the pressurization rate since bending segments encounter new contacts which limit deflection and therefore the bending stress developed in the fiber.

As the consolidation cycle continues, the number of contacts increases as neighboring fiber segments bend, and touch to form new contacts. When the density is low the effective contact stress is large but reduces to a value equal to the externally applied pressure as the relative density approaches unity. The majority of damage occurs therefore at the onset of consolidation. A micromechanical model can be developed to describe this process.

6.2. Micromechanical model development

To understand and rationalize the experimentally observed fiber damage trends a representative unit cell model was developed (depicted in Fig. 13) incorporating the dominant micromechanical features active in a single fiber segment in bending (viscoplastic contacts, matrix flow under the fiber segment, fiber bending stiffness increasing with increasing density). In subsequent work [23] the evolution of fiber bending and fracture will be simulated by viewing the densifying composite as a stochastic distribution of unit cells of different fiber lengths. Both the cell length distribution and the accumulated (macroscopic) fiber damage, as a function of the time during the consolidation cycle can be predicted by summing the fiber fracture probabilities of each unit cell.

The micromechanical response of a single cell can be represented by a simple spring–dashpot (Maxwell element) configuration like the one shown in Fig. 14 where the spring represents the elastic fiber and the dashpots represent the viscoplastic crossover contacts [24, 25].

The micromechanical response of the unit cell, as a function of a time dependent processing stress, is then described by four coupled, ordinary differential equations (these are derived in the Appendix)

\[ F(t) = k \varepsilon(t) \Delta t(t) \]

\[ \dot{\varepsilon}(t) = -B_0 \exp \left( \frac{-Q}{kT} \right) \frac{\sigma(t)}{\dot{\varepsilon}(t)} \]
where \( F(t) \) is the loading rate applied to the unit cell, \( a(t) \) and \( \dot{a}(t) \) are the matrix–matrix crossover contact area and its rate of change, \( r(t) \) and \( \dot{r}(t) \) are the stress developed at the crossover contact and its rate of change, \( z_0(t) \) and \( \dot{z}_0(t) \) are the matrix–matrix contact height and its rate of change, \( y(t) \) and \( \dot{y}(t) \) are the matrix–matrix contact length and its rate of change, \( h(t) \) is the rate of change of the overall unit cell height, \( k(t) \) and \( \dot{k}(t) \) are the elastic bending stiffness of the fiber segment which spans the contacts and its rate of change, and \( B_0, Q, n, p \) and \( d(t) \) have been defined previously.

Equations (9)-(12) can be simultaneously solved using a numerical ODE integration method (for example a Runga-Kutta solver). A program to solve the equations has been implemented using MATHEMATICA™ [26]. Simulation of the experimental process cycles discussed in Section 5.2 required about 2 min to complete on a 33 MHz 486 PC.

6.3. Fiber fracture simulation

The predictions of the unit cell model are given in Fig. 15(a–c) for two simulated process cycles, corresponding to the experimental 840°C isothermal cycles VHP-1 and 2. The viscoplastic response of the matrix alloy was determined by substituting the experimentally determined values of \( B_0, n, Q \) (Table 1) and the functional form of \( d(t) \) into equation (10). Each simulation was terminated at the same final relative density of 0.81. In each of the two simulations a unit cell 30 fiber diameters long \( (\ell = 30 d) \) was used. An initial contact height \( z_0(0) = 0.7 d \) \( (8.4 \text{ m}) \) represented the initial thickness of matrix material located between two crossing metallized fibers (we have assumed that the metal coating which surrounds each fiber is a uniform \( 4.2 \mu \text{m} \) in thickness for resultant matrix volume fraction of 0.65 and is consistent with the composite system studied here). An initial contact length of \( y_0(0) = 0.15 \ell \) \( (\ell = 30 d) \) was selected to obtain an initial cell density of 0.50 which was consistent with the initial density of the preconsolidated specimen. The density response of the unit cell simulation compared favorably to the experimentally measured densification response which indicates that the chosen initial conditions were physically reasonable.

First, consider the simulated densification response of the cell, Fig. 15(a). The model predicts a rapid rise in cell density due to VHP-2 and a more gradual increase in density due to VHP-1, consistent with experimental observations. Figure 15(b) shows that the rate at which the process stress is applied has a strong effect on the maximum fiber bending stress and ultimately fiber survivability [see Fig. 15(c)]. The VHP-1 process stress cycle is applied at a rate sufficiently slow that it allows the matrix to creep along the fiber axis without simultaneously subjecting the fiber segment to a bending stress sufficient to cause fracture. In contrast, the matrix creep strain is negligible the first 200 s of the VHP-2 simulation. In the VHP-2 simulation the unit cell densification is due almost entirely to fiber deflection [Fig. 15(a)]. The excessive deflections involved resulted in a very low fiber survivability which is consistent with experimental observations [Figs 7(c) and 8(c)].

6.4. Grain growth effects during consolidation

Significant matrix grain growth accompanies the extended thermal exposure of the consolidation process [13] and [see equation (1)] can influence the constitutive behavior. The steady state strain-rate, \( \dot{\epsilon} \), decreases as the time and temperature dependent grain size, \( d \), increases (for a constant stress) which would result in an increase in the creep resistance of the matrix and therefore an increased potential for fiber fracture. The process simulations conducted in Section 6.3 included this effect. The significance of not including concurrent matrix grain growth (i.e. keeping the grain size constant, \( d(t) = d_0 \)) on fiber survivability can be seen in Fig. 16. For each simulation the probability of fiber fracture decreased when grain growth was inhibited. The effect was more pronounced.

![Fig. 15. Micromechanical response of the unit cell when subjected to simulated process cycles VHP-1 and 2. (a) Predicted cell densification, (b) fiber bending stresses and (c) fiber survivability.](image-url)
6.5. Fiber strength degradation

The fiber reference strength, \( \sigma_0 \), can be significantly degraded, at the processing temperatures because of chemical interactions with the Ti-6Al-4V matrix alloy [5]. The significance can be assessed by repeating the two unit cell simulations of process cycles VHP-1 and 2 for a range of fiber reference strengths (\( \sigma_0, 0.90 \sigma_0, \) and 0.75 \( \sigma_0 \)) and comparing them in Fig. 17. In our analysis we have assumed that the damage occurs immediately during the process cycle (in reality it probably exhibits a time dependence that remains to be investigated). The simulations show that even a modest reduction in fiber strength can have a marked effect on the fiber survival probability. For example, reducing \( \sigma_0 \) by 10% changes the survivability from 50% to about 15% when the unit cell was processed by the VHP-2 cycle. The extent is very sensitive to the extent of the fiber reference strength degradation.

Fig. 16. The effect of inhibiting concurrent matrix grain growth for both VHP-1 and 2 simulations. Survivability is improved by inhibiting matrix grain growth.

in VHP-1 because of the longer high temperature exposure of this particular process cycle.

7. CONCLUSIONS

Individually metallized, continuous alumina fiber tows have low packing densities compared to the ideal hexagonal packing possible with parallel fibers of circular cross-section. To densify such a system, while avoiding fiber breaks, the process cycle must be designed to allow sufficient time for matrix creep alone to eliminate the void volume contained within the specimen. The work reported here has shown that this will only be satisfied if (1) the time required to transfer matrix material to the void space is within the time frame of the process cycle, (2) the required matrix strain rates are attainable at process stresses which do not cause excessive fiber deflection, (3) significant matrix grain growth is inhibited during processing and (4) fiber strength is retained during high temperature processing. Representative unit cell simulations have indicated that materials which exhibit high strain rates at low process stresses (i.e. enhanced superplastic behavior), are resistant to grain growth at the consolidation temperature and are inhibited from reacting chemically with the fiber reinforcement are likely to be preferred candidate matrices for composite fabrication.

Acknowledgements—The authors would like to thank Drs H. Deve, J. Storer, R. Kieschke and Mr P. Debruzzi of the 3M Metal Matrix Composites Center for their advice and assistance. We are grateful for support of this work by ARPA (Program Manager Mr W. Barker) through ONR grant N-00014-91-J-4089 (monitor Dr S. Fishman).

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APPENDIX

Derivation of the Approximate Constitutive Equations for the Unit Cell Micromechanical Response

The force–deflection relationship of the fiber span depicted in Fig. 13 is assumed to be linear elastic with a time dependent stiffness

\[ F(t) = k(t)\Delta r(t) \]  

(A1)

where \( F(t) \) is the force deflecting the fiber, \( k(t) \) the fiber bend stiffness and \( \Delta r(t) \) is the maximum fiber deflection. The fiber elastic constant, \( k(t) \), is obtained from simple beam theory for a statically determinate beam, of circular cross-section, with fixed ends

\[ k(t) = \frac{3\pi E_d d(t)}{(A(t))} \]  

(A2)

where \( l(t) \) is the instantaneous fiber span length, and \( d(t) \) and \( E_d \) are the fiber diameter and temperature dependent fiber Young's modulus respectively. In the metalized NEXTEL 610™ composite system the fiber is completely coated by matrix material thereby increasing the bending stiffness of the segment. The bending of such a composite beam is difficult to analyze, but for thin metal coatings whose Young's modulus (at elevated temperature) is significantly lower than that of the fiber, bending will be dominated by the stiffness of the fiber span. Here, the effect of the matrix coating is therefore ignored resulting in a slightly conservative estimate of the fiber bending stress.

The cell has a constant length, \( l_{\text{cell}} \), and a width equal to a single fiber diameter, \( d_f \), such that the total cell area is

\[ A = l_{\text{cell}}d_f. \]  

(A3)

The cell length, \( l_{\text{cell}} \), is the sum of the lengths of the two, time-dependent, matrix–matrix contacts, \( 2y_c[t] \), and the length of the fiber segment spanning the void space between the contacts, \( l(t) \)

\[ l_{\text{cell}} = l(t) + 2y_c[t]. \]  

(A4)

The length of the fiber segment spanning the contacts follows from equation (A4)

\[ l(t) = l_{\text{cell}} - 2y_c[t]. \]  

(A5)

Substituting equation (A5) into equation (A2) yields the time dependent, elastic bend stiffness of the fiber segment

\[ k(t) = \frac{3\pi E_d l(t)}{(l_{\text{cell}} - 2y_c[t])}. \]  

(A6)

Applying a time dependent consolidation pressure, \( \sigma(t) \), to the unit cell produces a force, \( F(t) \), equal to

\[ F(t) = a(t)e(t) \]  

(A7)

which compresses the cell and deflects the fiber segment. The force is transmitted through the upper viscoplastic contact and a force balance requires

\[ F(t) = a(t)e(t)v(t) \]  

(A8)

where \( a(t) \) is the contact flow stress (assumed to be that of a uniaxial compression test) and \( v(t) \) is the instantaneous area of the upper contact equal to

\[ v(t) = d_fy_c[t]. \]  

(A9)

The interpenetration of two metal coated fibers at a crossover contact is significantly more complex than the idealization above. The underlying mechanics of the problem have been retained however in the modeling approach: (1) viscoplastic contact deformation behavior and (2) metal flow from the contact area resulting in densification. The time derivative of equation (A8) is the loading rate applied to the cell

\[ \frac{dF}{dt} = a(t)e(t)v(t) + a(t)\sigma(t) \]  

(A10)

When pressure is applied to the specimen, densification is due to the combined mechanisms of fiber deflection, time-independent plasticity (plastic flow) and inelastic deformation (transient and steady state creep) at the metalized fiber contacts. Here, only the mechanisms of elastic fiber deflection and matrix steady state creep are assumed operative. The mechanisms of plastic flow and transient creep effectively reduce the creep resistance of the contacts for a fixed cell loading rate so our assumption is likely to somewhat overestimate the probability of fiber fracture.

We have assumed that the contact compressive strain rate can be represented by the constitutive law which describes the steady state deformation behavior of the matrix [see text, equation (1)]. The strain rate of the contact, under the influence of \( \sigma(t) \), then becomes

\[ \dot{\epsilon}_c = B_0\exp\left(-\frac{Q}{RT}\right)\frac{\sigma(t)}{\dot{\epsilon}_c}\]  

(A11)

where \( \dot{\epsilon}_c \) is the true strain rate of the contact in the z-direction (the remaining parameters having previously been defined). Equation (A11) can be rewritten as a function of \( z_c[t] \), the instantaneous height of the contact, using the relationship

\[ \dot{z}_c = \frac{\dot{z}_c}{z_c} \]  

(A12)

Equating equations (A11) and (A12) and rearranging yields

\[ \dot{z}_c = B_0\dot{\epsilon}_c\exp\left(-\frac{Q}{kT}\right)\frac{\sigma(t)}{\dot{\epsilon}_c}. \]  

(A13)
The strain rate predicted by equation (A13) is negative under the influence of a compressive contact stress. By conservation of mass and assuming that the contact deforms by plane strain (strain in the \( y-z \) plane only) the contact strain rate in the \( y \)-direction is equal and opposite in sign to the contact strain rate in the \( z \)-direction

\[
\dot{\varepsilon}_y = - \dot{\varepsilon}_z \quad \text{(A14)}
\]
or

\[
\dot{y}[t] = - \frac{\delta y[t]}{\delta z[t]} . \quad \text{(A15)}
\]

The instantaneous cell height, \( h[t] \), can be obtained by inspection of the Maxwell element model in Fig. 14:

\[
h[t] = 2z[t] + d - \Delta h[t] . \quad \text{(A16)}
\]

Solving for \( \Delta h[t] \) in equation (A1) and substituting into equation (A16) yields

\[
h[t] = 2z[t] + d - \frac{F[t]}{k[t]} . \quad \text{(A17)}
\]

The time derivative of equation (A17) is the cell height velocity at a time \( t \) during the consolidation cycle

\[
\dot{h}[t] = 2\dot{z}[t] - \frac{\dot{F}[t]}{k[t]} + \frac{\dot{F}[t]}{(k[t])^2} \dot{h}[t] . \quad \text{(A18)}
\]

To solve for the response of the unit cell, the cell force, \( F[t] \), is first determined from a given pressurization cycle, \( t \). With the derivatives \( f[t] \), \( z[t] \), \( y[t] \) and \( h[t] \) given by equations (A10), (A13), (A15) and (A18) respectively, the problem reduces to determining the appropriate initial conditions \((F[0] = 0, \Delta h[0] = 0.8 \ \mu \text{m}, \ z[0] = 0.7 \ \text{m}, \ y[0] = 0.15 \ \text{m}, \ h[0] = 1.7 \ \text{m}, \ \text{see Section 6.3}) and simultaneously solving a system of four first order, ordinary differential equations for \( F[t], \ z[t], \ y[t] \) and \( h[t] \). Once the time-dependent variables are numerically determined (using a Runge-Kutta, ODE integration method), the fiber bending stress, \( \sigma[t] \), can then be evaluated using

\[
\sigma[t] = 4 \pi \frac{F[t][d][t]}{d} . \quad \text{(A19)}
\]

which is the maximum tensile bending stress in a beam of circular cross-section with fixed ends [27]. The survivability of the fiber segment, \( \Psi[t] \), can then be determined, as a function of the process cycle, using the Weibull relationship [28]

\[
\Psi[t] = \exp \left[ - \kappa \frac{h[t]}{l} \left( \frac{\sigma[t]}{\sigma_0} \right)^{m} \right] \quad 0 \leq \Psi[t] \leq 1 \quad \text{(A20)}
\]

where \( m \) is the Weibull modulus of the fiber, \( \sigma_0 \) is the reference strength at some reference length \( l_0 \) and \( \kappa \) is the load factor which is a measure of the uniformity of the stress distribution in a bending beam of circular cross-section [29]. When \( \Psi[t] \) is equal to unity the probability of fiber fracture is zero. The value of \( \kappa \) is a function solely of the Weibull modulus and is equal to

\[
\kappa = \frac{1}{2 \sqrt{\pi}} \left( \frac{m + 1}{2} \right) \quad \text{(A21)}
\]

where \( \Gamma \) denotes the Gamma function [30].